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REPORT NO. RS-TR-66-5

**METALLURGICAL EXAMINATION OF BULLPUP "A"
PROPULSION UNIT S/N 4800 AFTER THREE YEARS OF STORAGE**

by
J. T. Davidson

April 1966

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28 April 1966

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**METALLURGICAL EXAMINATION OF BULLPUP "A"
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J. T. Davidson

DA Project No. 1X242403D231

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**Materials Engineering and Development Branch
Structures and Mechanics Laboratory
Research and Development Directorate
U. S. Army Missile Command
Redstone Arsenal, Alabama 35809**

ABSTRACT

A tactical BULLPUP "A" prepackaged, storable, liquid propulsion unit was metallurgically examined after three years of storage. The examination was conducted relative to the storage requirements of the LANCE missile system (currently being developed by the U.S. Army Missile Command, Huntsville, Alabama) which utilizes materials and propellants similar to those employed in the BULLPUP "A."

The purpose of the evaluation was to acquire "real-time" storage data based on long periods of exposure.

The examination revealed that containment of MAF-1 fuel and inhibited red fuming nitric acid oxidizer by 2014-T6 aluminum alloy tankage for more than three years had not affected the mechanical, physical, or chemical properties of the materials to a degree that would affect tactical performance.

ACKNOWLEDGMENT

The author wishes to express his appreciation to Mr. Le Sumner for his valuable assistance in performing the metallography for the evaluation and also to Mr. Robert Cannon for performing the stress-free machining and sectioning operations that were responsible for the successful residual stress analysis of the unit.

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1. Introduction

The propulsion unit from the BULLPUP "A", a prepackaged, liquid propulsion unit, was examined after three years and four months of storage.

The objective of the examination was to determine the extent of corrosion that might have occurred in areas having contact with propellants, at points of dissimilar metals contacts, and at weld joints.

The examination was performed under the auspices of the LANCE Project Office, U.S. Army Missile Command (AMICOM), Redstone Arsenal, Alabama, as a part of the LANCE Storage and Sludging Program, a study to determine the effects of extended storage on the functional properties of LANCE materials.

Use of the BULLPUP unit in the LANCE Storage and Sludging Program was initiated and warranted because of the basic similarity of the two propulsion systems. Both the LANCE and BULLPUP propulsion units are of tandem-tank configuration and both employ integrally welded 2014-T6 aluminum alloy primary construction material. In addition, the propellants of the two units are inhibited red fuming nitric acid (IRFNA)/mixed amine systems, and are prepackaged, i.e., the tanks serve as hermetically sealed storage containers for the propellants.

The defueling operation and the analysis of propellants (fuel and oxidizer), sludge, etc. are the subjects of a separate study conducted by the Propulsion Laboratory, AMICOM, and will be reported in detail in another report. However, the fuel and oxidizer analyses are noted in this report because they contribute to the metallurgical analysis.

The unit was sectioned for internal examination by the Structures and Mechanics Laboratory, AMICOM, as a part of the Residual Stress Analysis Program. The residual stress information is discussed briefly in this report, and will be reported in detail in another report. Photographs taken during the sectioning operation are included in this report to show the sectioning techniques employed.

2. Procedure

The propulsion unit utilized for this examination was from BULLPUP "A" (LR58-RM-4), the Navy AGM-128 air-to-surface guided missile. Specifically, the LR58-RM-4 propulsion unit, serial number (S/N) 4800, assembly number 313207, from lot number 4, manufactured in January 1962 was supplied by the U.S. Naval Propellant Plant, Indian Head, Maryland. The unit was delivered to the Materials Engineering and Development Branch, Structures and Mechanics Laboratory, AMICOM, after the oxidizer, fuel, and solid propellant gas generator (SPGG) grain had been removed.

The internal surfaces of the oxidizer tank were examined for deposits, and two types of deposits were observed: an amber deposit on the tank walls, and a heavier, thicker, green deposit adhering loosely to the gas diffuser and deflector assembly. These deposits were collected separately and subjected to chemical analysis.

The internal surface of the oxidizer tank was optically examined for dissolution corrosion effects.

The thickness of the oxidizer tank walls was measured and recorded.

An example of each weld and weld type in the oxidizer tank sub-assembly was mounted and examined microscopically.

3. Discussion

The preloading propellant analysis and the loading and sealing of the propulsion unit was accomplished in January 1962. The propellants were removed from the unit in May 1965, making a total storage time (including the transport time from Indian Head to Redstone Arsenal) of three years and four months. The storage requirements for the system were -80° to $+160^{\circ}\text{F}$.

a. Propellant Analysis

A chemical analysis of the propellants was made in January 1962 at the time of loading and sealing. Then in May 1965, after three years and four months of storage, the propellants were removed from the unit and analyzed again. A comparison of the two analyses is given below:

IRFNA

18 Jan 1962

HNO ₃ (%)	83.16
NO ₂ (%)	14.05
HF (%)	0.60
Total solids (%)	0.01
H ₂ O (%)	2.18

28 May 1965

HNO ₃ (%)	83.00
NO ₂ (%)	14.19
HF (%)	0.52
Total solids (%)	0.28
H ₂ O (%)	--

IRFNA met the specification requirements of MIL-P-72554E Type III A acids.

MAF-1

18 Jan 1962

DETA (%)	50.27
UDMH (%)	40.38
ACN (%)	9.0
H ₂ O (%)	0.35

22 Jun 1965

DETA (%)	50.0
UDMH (%)	39.35
ACN (%)	9.3
H ₂ O (%)	--

The UDMH was approximately 1.0 percent low, according to RMD Specification No. 4034.

A note of interest in the chemistry of the IRFNA is the apparent stability of the HF after 3.3 years of storage. The drop from 0.60 percent weight in 1962 to 0.52 percent weight in 1965 is almost within experimental error limits, and is particularly impressive when compared to other aluminum/IRFNA systems such as the 6000 series aluminum alloys which indicate HF losses of 80 to 90 percent in five years of storage. This would seem to indicate that the phenomenon of passivation of aluminum by HF (in which the HF slowly forms a fluoride passive coating on the aluminum) is less pronounced with the 2014-T6 aluminum alloy of the BULLPUP and LANCE missiles than with the 6061-T6 aluminum alloy of the LARK and SPARROW missiles. The fuel and oxidizer were drained from the unit through a 10-14M (micron) filter. The fuel filter did not display sufficient residue to warrant an analysis; however, residue on the oxidizer filter was quite heavy and was analyzed.

b. Residue Analysis

Two separate residues (deposits) were identified by color; one was white and the other, green. Semi-quantitative spectrographic analysis of the white deposit indicated 8.3 percent sodium, 18.0 percent silicon, 5.6 percent calcium, and 3.3 percent aluminum.

Analysis of the green deposit indicated 17.0 percent iron, 6.6 percent aluminum, 3.8 percent chromium, 0.71 percent nickel, and 0.23 percent manganese.

The dry deposits removed from the oxidizer tank after sectioning were analyzed and the results indicated that:

- 1) The amber deposits on the oxidizer tank walls and the white deposit taken from the IRFNA were the same material.
- 2) The green flake deposits taken from the SPGG diffuser and deflector and the green deposits taken from the IRFNA were the same material.

The tankage was decontaminated after draining in order to preserve the storage-induced fluoride passive coating on the interior surface. To this end, a long-term, low-pressure, warm, nitrogen-gas purge was employed. The adherence of the lightly attached amber deposit on the oxidizer tank after decontamination indicates that the decontamination procedure was successful.

c. Residual Stress Evaluation

Sectioning of the propulsion unit was performed as a part of the residual stress evaluation. The stress evaluation was of particular importance since the stress-corrosion characteristics of the 2014-T6 aluminum/IRFNA couple determine the ability to achieve long-term storage. Long-term storage is one of the primary areas where the state-of-the-art is long extended in the development of packaged liquid propulsion systems. Only limited experience data is available on the compatibility of the materials utilized in the BULLPUP and LANCE systems. The evaluation of the stress-corrosion characteristics of the BULLPUP propulsion unit provided data based on a long period of storage. Such "real-time" data is necessary for meaningful results and correlation of data to the LANCE design.

Stress corrosion is a complex interaction of corrosive attack with sustained tension stress at the surface of the metal that causes cracking. In the stress-corrosion cracking process there is a greater and faster deterioration in mechanical properties through simultaneous action of static tension stress and corrosion than would occur as a result of the separate but additive effects of the phenomena.

Processing techniques for both the BULLPUP and LANCE propulsion systems require the use of "as-welded" 2014-T6 aluminum alloy structures. That is, the structures are utilized without a post-assembly or postwelding, stress-relief treatment. As a consequence stresses introduced into the structures by the general assembly and welding processes remain in the structure. Of the two modes of stress, the induction stresses (those stresses induced in and by welding) are of basic importance to the stress-corrosion and long-term storage evaluation.

The mechanics of stress induction during welding indicates that heat softens the metal adjacent to the weld and causes thermal expansion of this material. Cooler metal away from the weld restrains the thermal expansion and introduces compressive stresses parallel to the weld that are great enough to cause plastic deformation. On cooling, the heated region contracts and the yield strength increases. Tensile stresses are thus introduced.

As can be seen, the role of stress in promoting stress-corrosion cracking is important. In a residual stress analysis, this is directly related to the stress-corrosion characteristics of a structural unity. Accurate determination of structural residual stress uninfluenced by sectioning or analytical processing is equally important. Consequently, great care was taken in the cutting and sectioning operation. The gentle nature of the cutting can best be illustrated by the fact that a 0.90-inch steel roll pin, used as a location pin for the nozzle to fuel tank thrust chamber couple, was able to withstand, without failure, all the circumferential forces applied during the lathe cutting of the unit. Photographs taken during various phases of the cutting operation are included as part of this report.

The relaxation technique was employed in the residual stress analysis. Seventy-two rosette-type strain gages were attached in areas of primary stress induction sites. Strain gage variations before and after cutting were used to calculate the residual figures. The predominant stress in the structure was a 12,000 psi tensile stress on the outer surface of the fuel tank wall at the center bulkhead weld.

d. Examination of Interior Surfaces

Metallographic examination of the interior surfaces of the fuel tank did not reveal any form of corrosive attack. Surfaces of the 2014-T6 aluminum alloy tank were bright, clean, and smooth.

Examination of the interior surfaces of the oxidizer tank revealed, in addition to the amber colored deposit, a slight etch condition over all surfaces. This etch was slightly more apparent at the weld heat affected zone (HAZ). The surface of the stainless steel SPGG diffuser and deflector was etched fairly heavily under the green flaky deposit.

No evidence of detrimental intergranular corrosion, galvanic corrosion, or stress corrosion was found in any area of the propulsion unit.

Incidental to this evaluation, but nevertheless important to the storage life, was the good condition of the elastomeric "O" rings.

e. Wall Thickness

The wall thickness of the propulsion unit oxidizer tank after three years and four months of storage was measured and found to be within drawing requirements. A pictorial record of the evaluation of the BULLPUP propulsion unit, including photomicrographs of representative microstructures, is included as part of this report.

4. Conclusion

After 3.3 years of storage the mechanical, physical, and chemical properties of the 2014-T6 aluminum alloy used in the construction of the BULLPUP "A" (LR58-RM-4) propulsion unit, assembly number 313207, S/N 4800 experienced no significant degradation.

Residue found in the oxidizer tank was caused by a reaction between the stainless steel SPGG diffuser and deflector assembly and the IRFNA.

Results of this evaluation support utilization of 2014-T6 aluminum alloy for the LANCE system relative to extended storage requirements and materials compatibility.

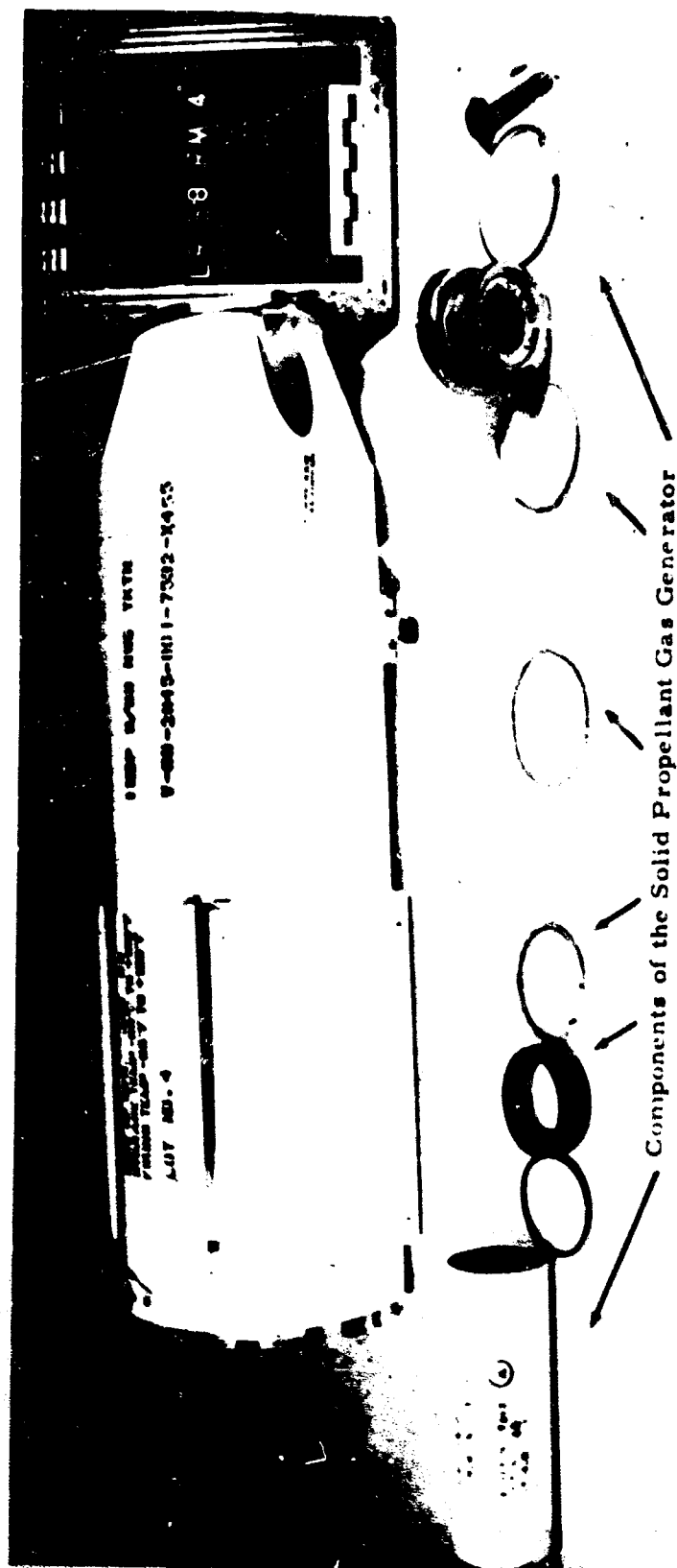
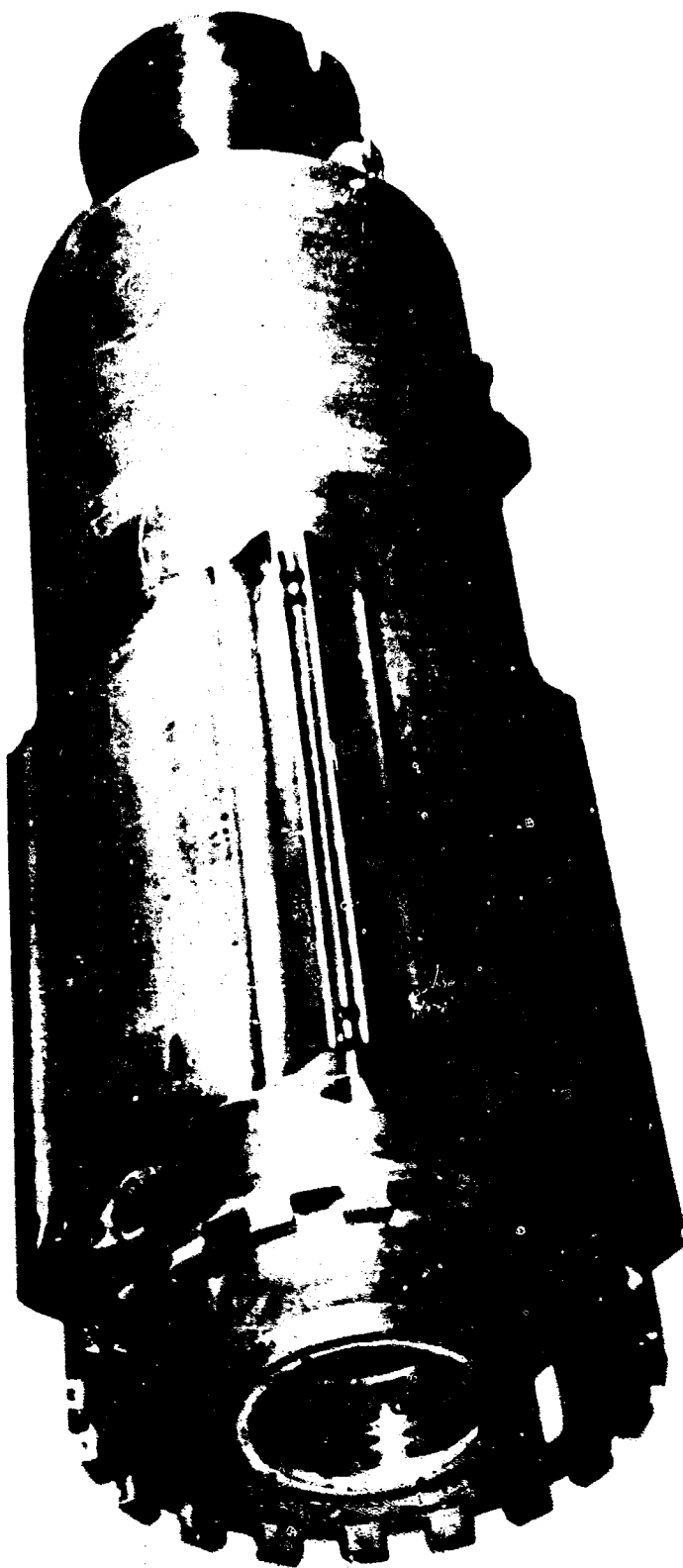
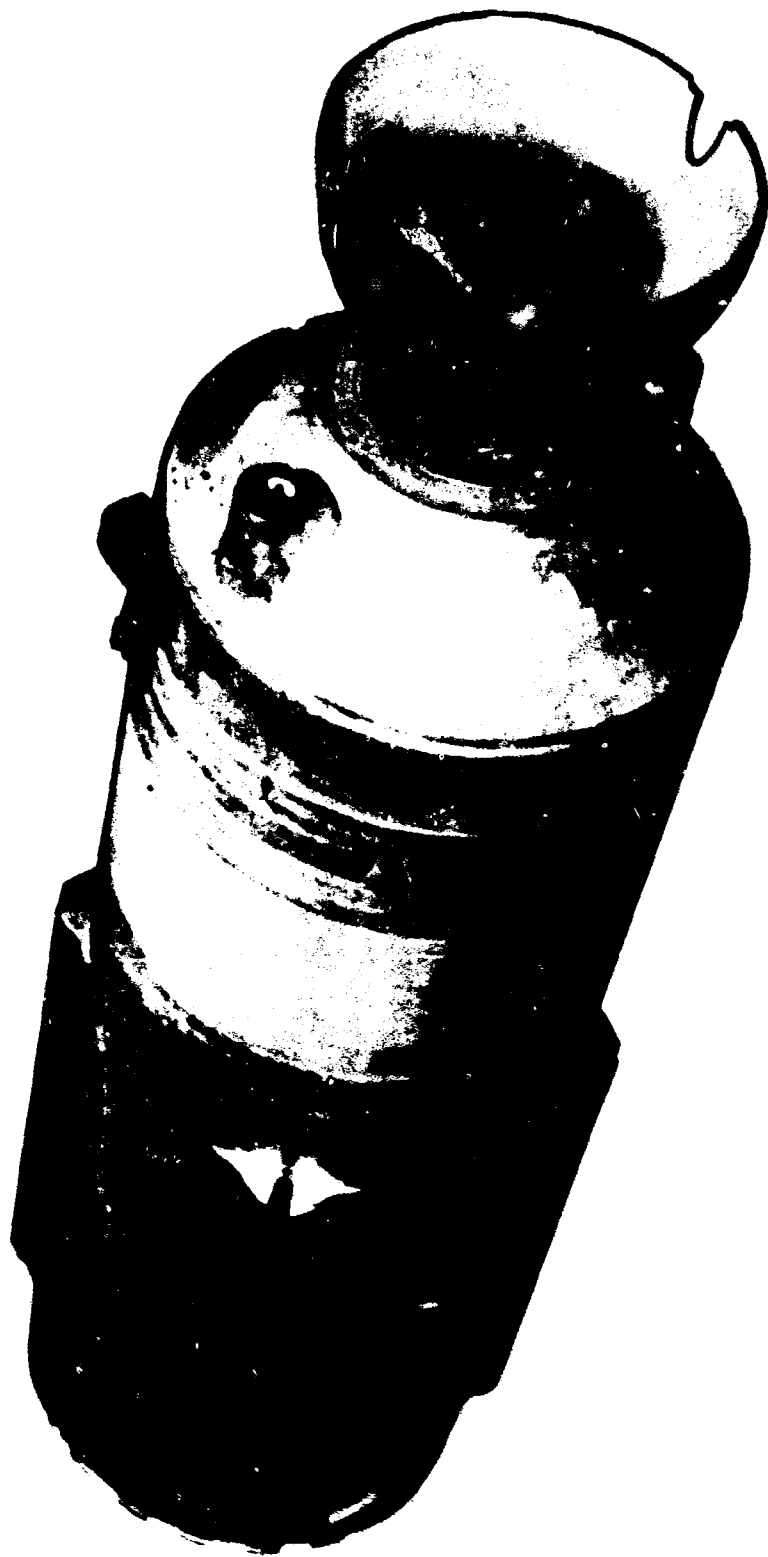


Figure 1. BULLPUP "A" Prepackaged Liquid Propulsion Unit S/N 4800



Forward End

Figure 2. Propulsion Unit S/N 4800 After Removal of Fairing Cone and Exterior Paint, Forward View



Aft End

Figure 3. Propulsion Unit S/N 4800 After Removal of Fairing Cone and
Exterior Paint, Aft View

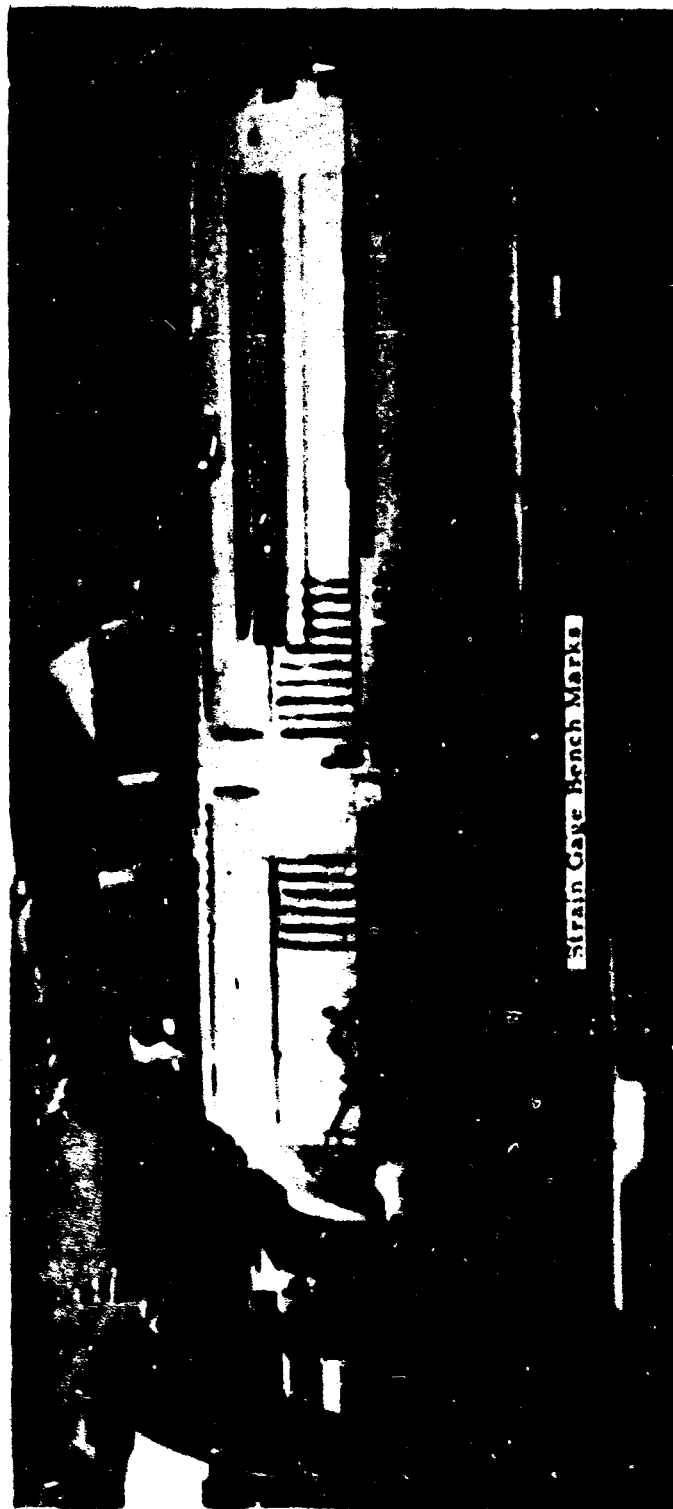


Figure 4. Propulsion Unit Installed in Lathe for Residual Stress Analysis Cutting

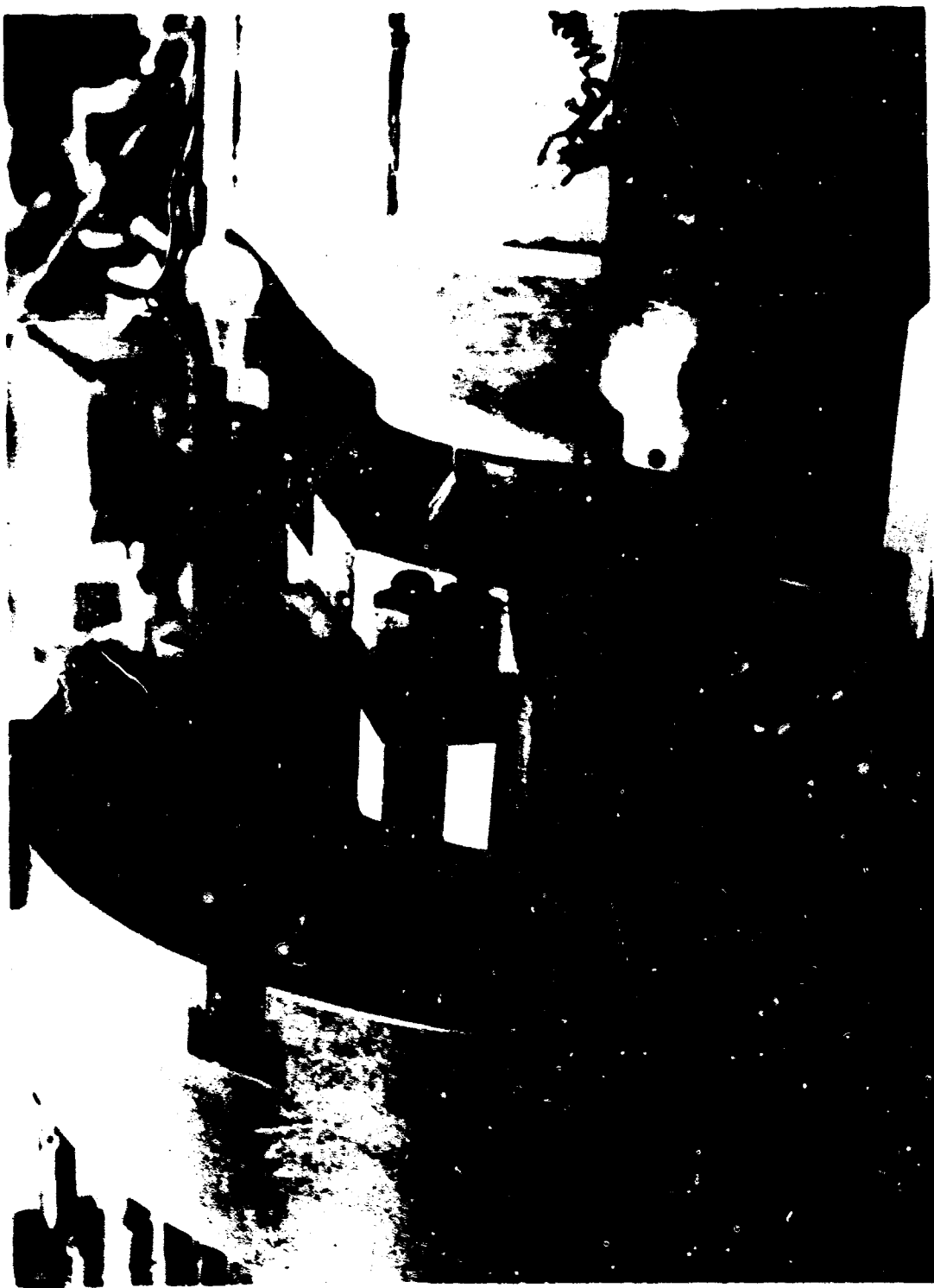


Figure 5. Low Stress Drive Fixture



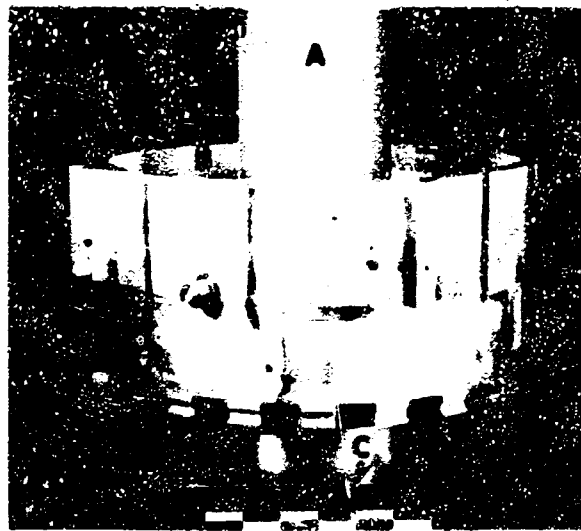
Figure 6. Low Stress Steady-Rest Tail-Stock Fixture



Figure 7. Residual Stress Strain Gage Installation, General View



Figure 8. Typical Strain Gage Installation, Three Separate Gages



- A. SPGG Tube
- B. Oxidizer Tank Wall
- C. Part of Cutting Fixture

Figure 9. Forward Section of Oxidizer Tank After First Cut



- A. Stainless Steel Solid Propellant Gas Diffuser and Deflector
- B. Aluminum SPGG Tube
- C. Aluminum Oxidizer Tank Wall

Figure 10. Deposits on Interior of Oxidizer Tank



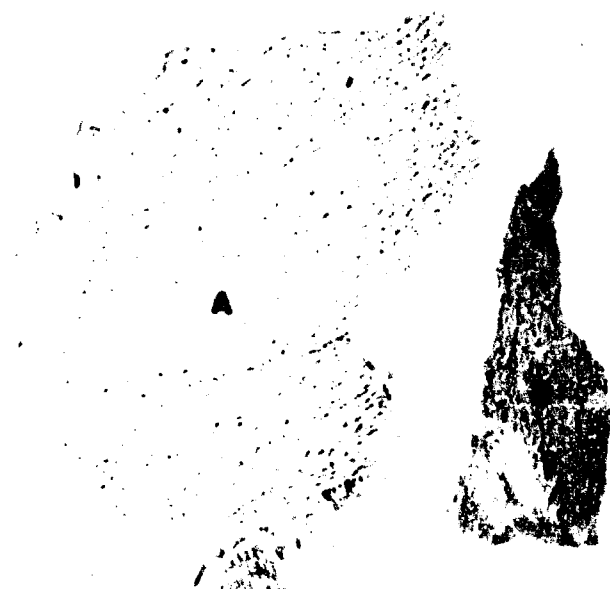
Deposit was lightly attached.

Figure 11. Deposit on Aluminum SPGG Tube



Deposit was lightly attached.

Figure 12. Deposit on Oxidizer Tank Solid
Propellant Gas Diffuser and Deflector



- A. Surface of Deposit, Light Green
- B. Underside of Deposit, Dark Green

**Figure 13. Deposits from Oxidizer Tank Solid
Propellant Gas Diffuser and Deflector**

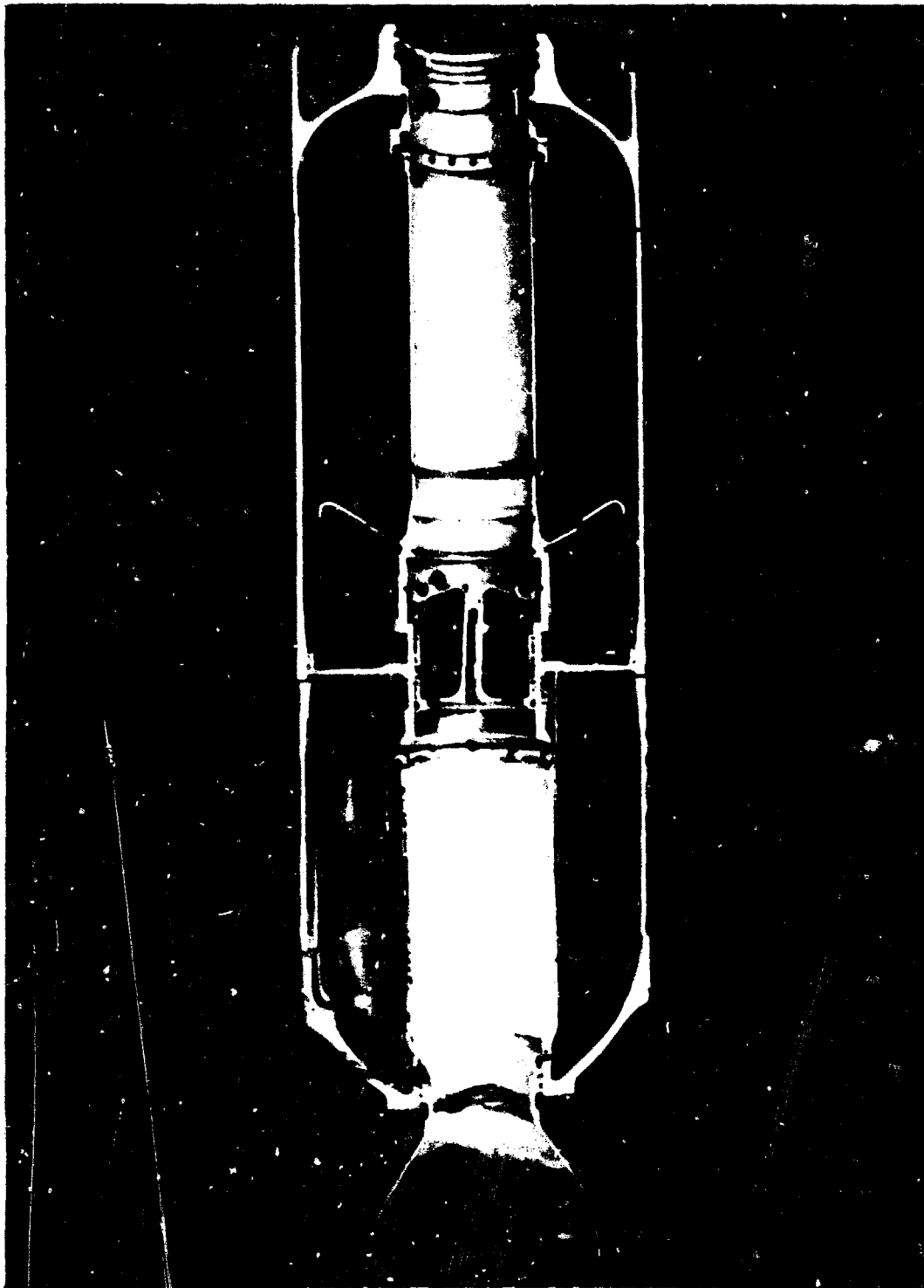
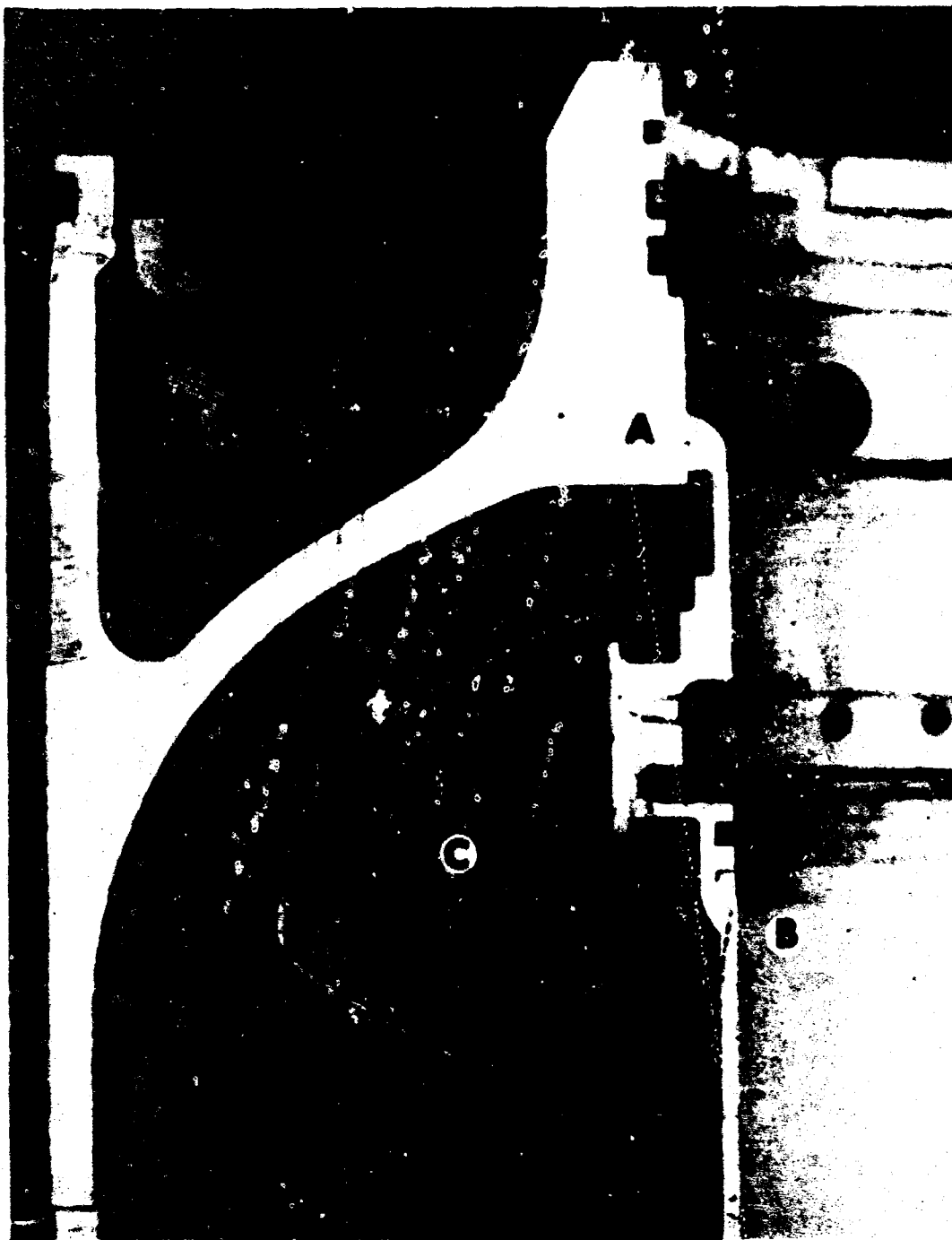


Figure 14. BULLPUP Propulsion Unit S/N 4800 After Three Years of Storage, Sectioned View



- A. Burst Band to Forward Dome Weld
- B. Burst Band to SPGG Tube Weld
- C. Solid Propellant Gas Diffuser and Deflector Assembly

Figure 15. Forward End of Oxidizer Tank, Sectioned, Close-Up View



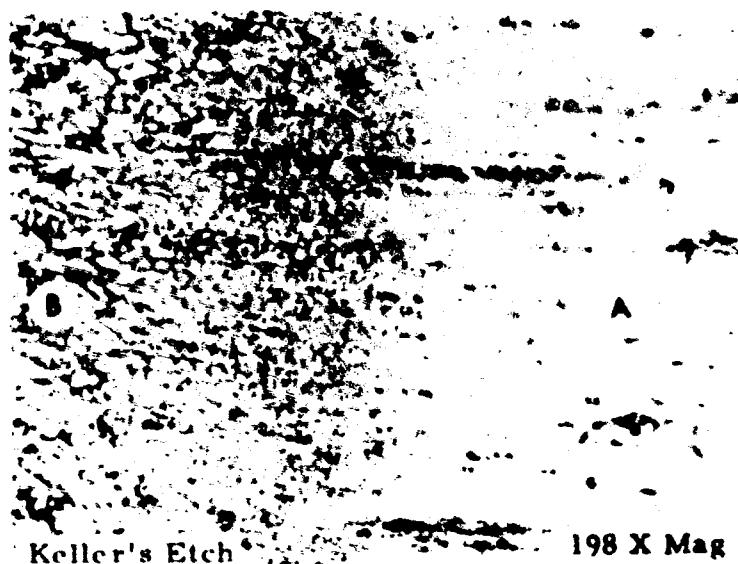
- A. Burst Band to Forward Dome
- B. Burst Band to SPGG Tube

Figure 16. Forward Dome Weldments



- A. Aluminum Base Metal (Dome)
- B. Weld Metal

Figure 17. Burst Band to Forward Dome Weld Fusion Zone



Keller's Etch 198 X Mag

A. Aluminum Base Metal (Burst Band)
 B. Weld Metal

Figure 18. Burst Band to SPGG Tube Weld Fusion Zone



Keller's Etch 198 X Mag

A. Aluminum Base Metal (SPGG Tube)
 B. Weld Metal
 C. IRFNA Contact Surface

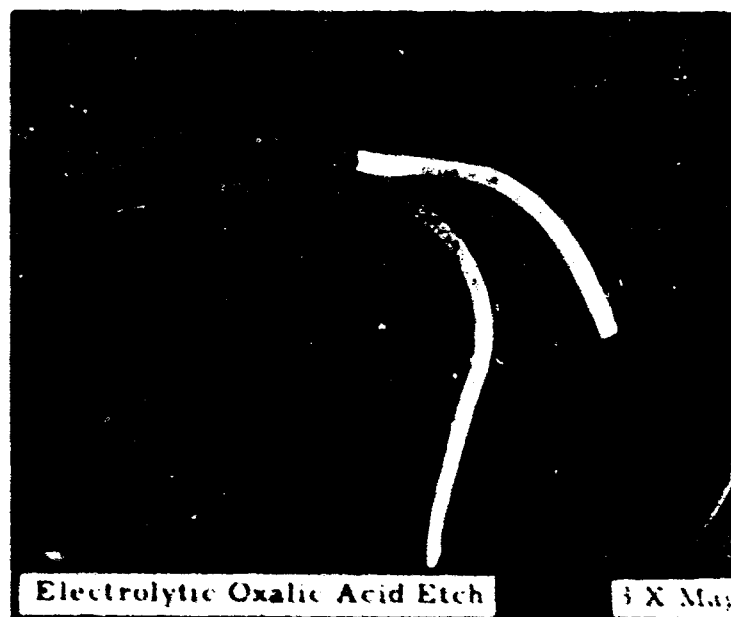
Figure 19. IRFNA Contact Surface at Burst Band to SPGG Weld HAZ



Keller's Etch 198 X Mag

- A. Aluminum Base Metal (Burst Band)
- B. IRFNA Contact Surface

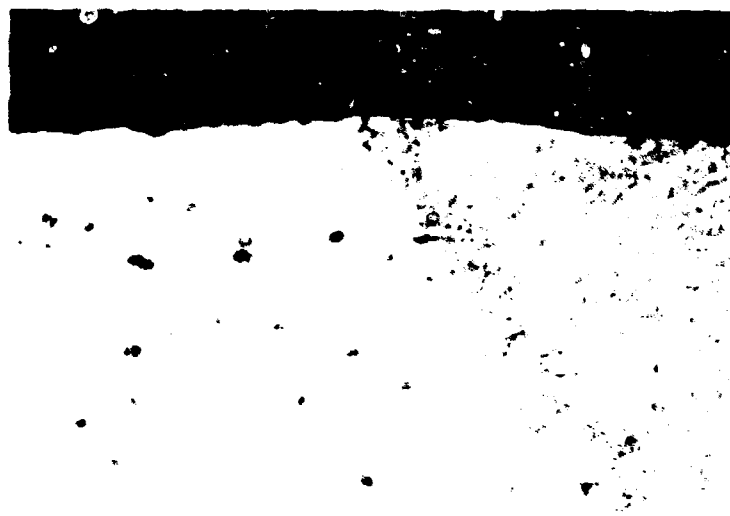
Figure 20. IRFNA Contact Surface at Burst Band to SPGG Tube Weld HAZ



Electrolytic Oxalic Acid Etch

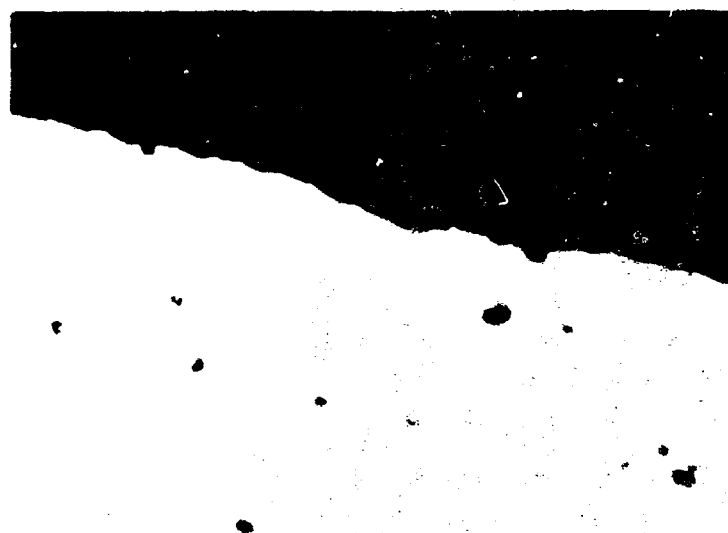
3 X Mag

Figure 21. Stainless Steel Solid Propellant Gas Diffuser and Deflector, in Two Pieces



Electrolytic Oxalic Acid Etch 198 X Mag

Figure 22. Stainless Steel Solid Propellant
Gas Diffuser and Deflector Weld



Electrolytic Oxalic Acid Etch 396 X Mag

Figure 23. Stainless Steel Solid Propellant
Gas Diffuser and Deflector Surface After
Removal of Corrosion Deposits



- A. Oxidizer Tank Baffle
- B. Baffle to SPGG Tube Weld
- C. SPGG Tube to Shear Slide Housing Weld
- D. Oxidizer Orifice
- E. Fuel Tank Burst Band and Bulkhead to Fuel Tank Baffle Weld
- F. Oxidizer Director and Screen Assembly
- G. Center Bulkhead to Oxidizer Tank Shell Weld
- H. Fuel Tank Solid Propellant Gas Diffuser and Deflector

Figure 24. Center Bulkhead Interface, Sectioned View

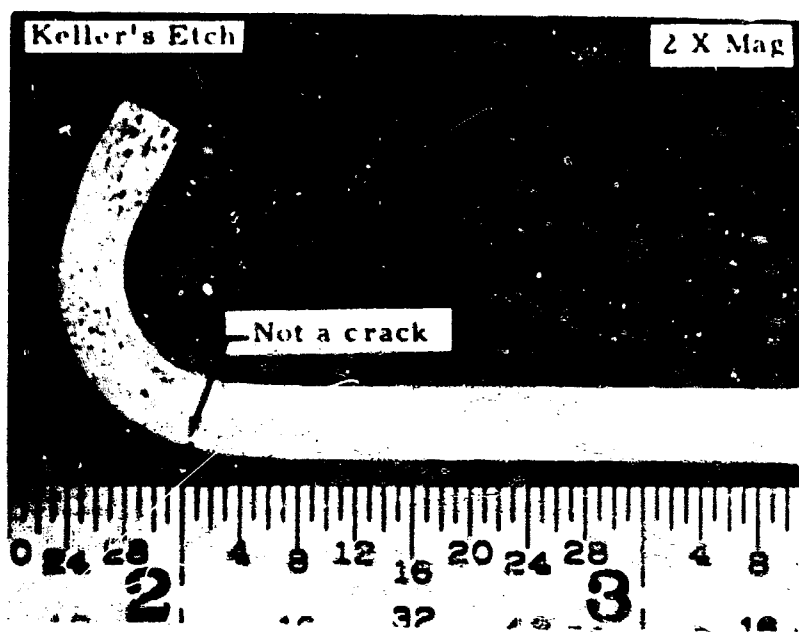
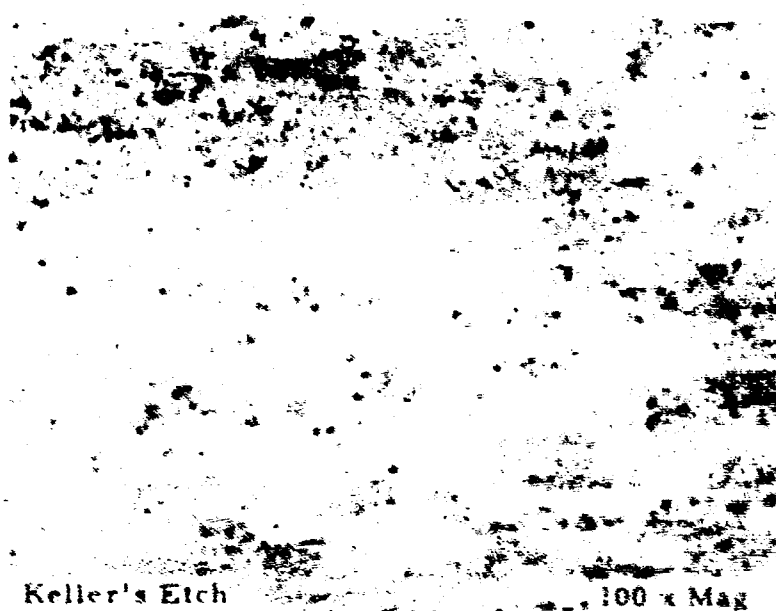
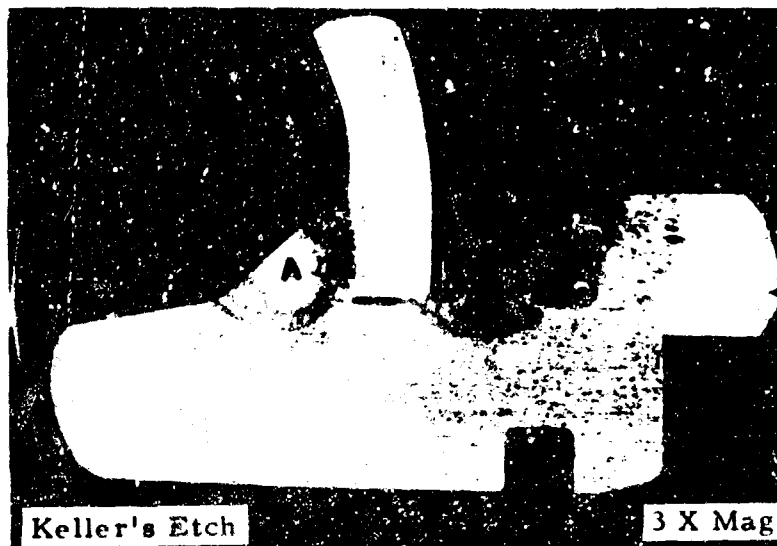


Figure 25. Aluminum Oxidizer Baffle

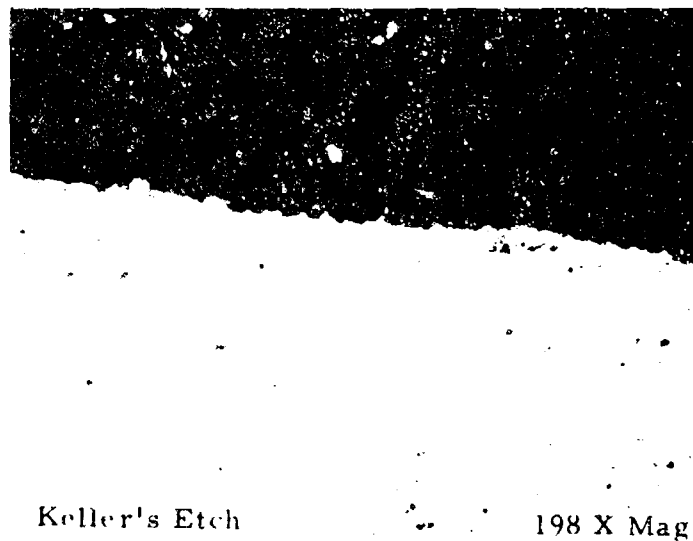


Grain exposure unaffected by contact.

Figure 26. Aluminum Oxidizer Baffle,
Grain Structure



A. Oxidizer Baffle to SPGG Tube Weld
 B. SPGG Tube to Shear Slide Housing Weld
 Figure 27. Aluminum Oxidizer Baffle Welds

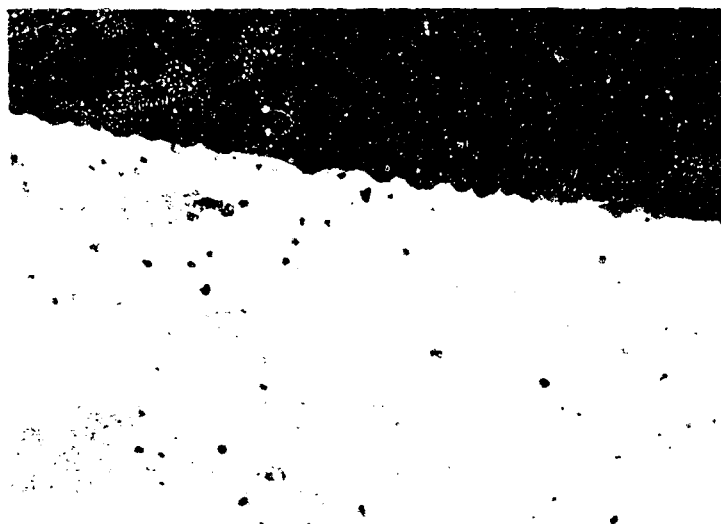


IRFNA corrosion was most severe at the HAZ surfaces.
 Figure 28. IRFNA Contact Surface of SPGG Tube HAZ at Oxidizer Baffle Weld

Weld Metal

Keller's Etch 198 X Mag

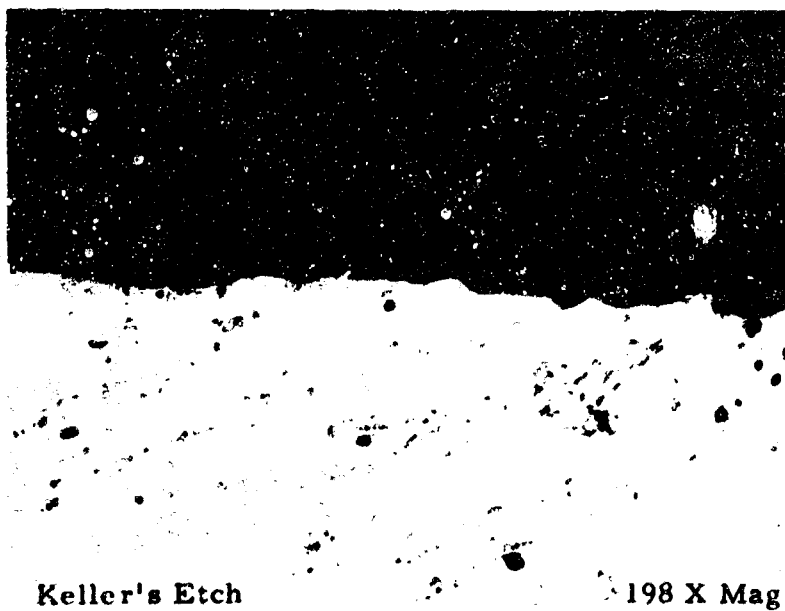
Figure 29. Aluminum Baffle to Stainless Steel SPGG Tube Weld Fusion Zone



Keller's Etch 198 X Mag

Degree of corrosion is the same in the HAZ of other aluminum components. Angularity of exposed grains does not appear to influence the amount of corrosion.

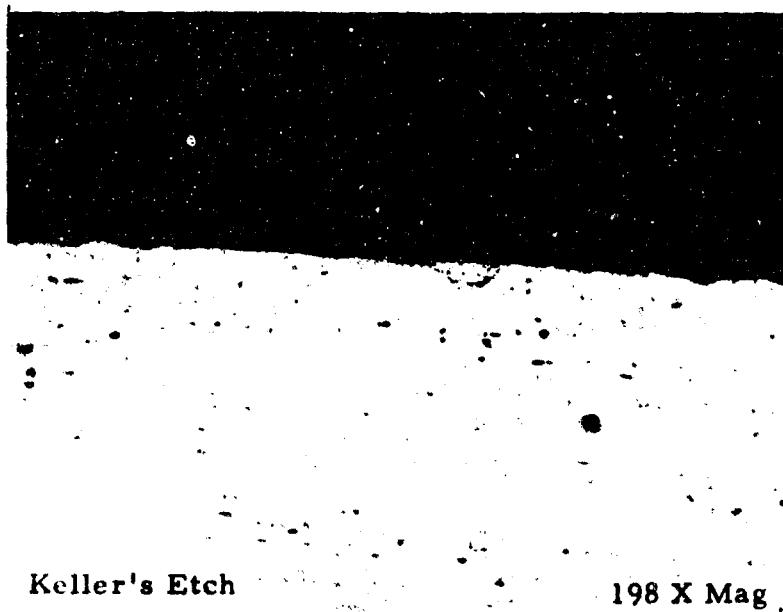
Figure 30. IRFNA Contact Surface of Aluminum Baffle HAZ at Stainless Steel SPGG Tube Weld



Keller's Etch

198 X Mag

Figure 31. IRFNA Contact Surface of Aluminum Shear Slide Housing HAZ at SPGG Tube Weld

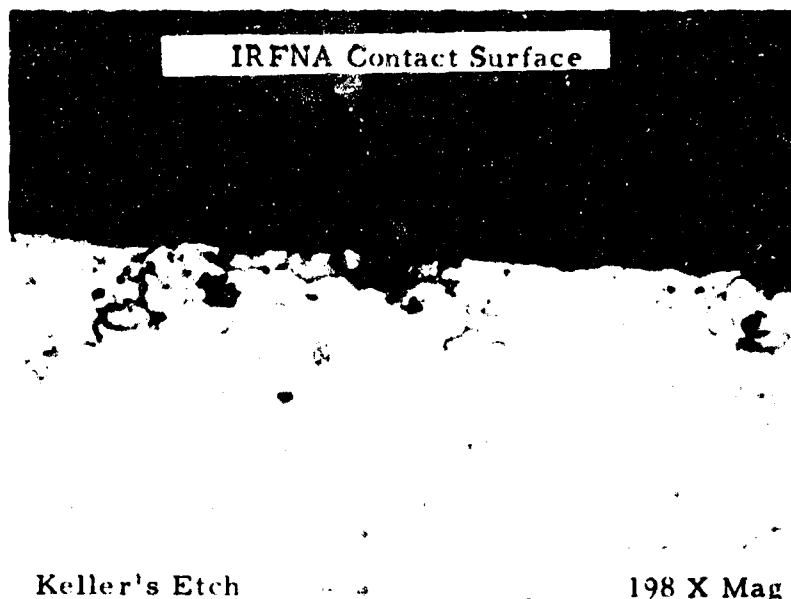


Keller's Etch

198 X Mag

Surface generally free of attack.

Figure 32. Interior Surface of Aluminum SPGG Tube That Had No IRFNA Contact



Intergranular attack and general surface dissolution corrosion have progressed further than any nitric-induced corrosion did during the 3.3 years of storage.

Figure 33. Same Surface as Shown in Figure 32
After Three Months of Exposure to Laboratory
Atmosphere

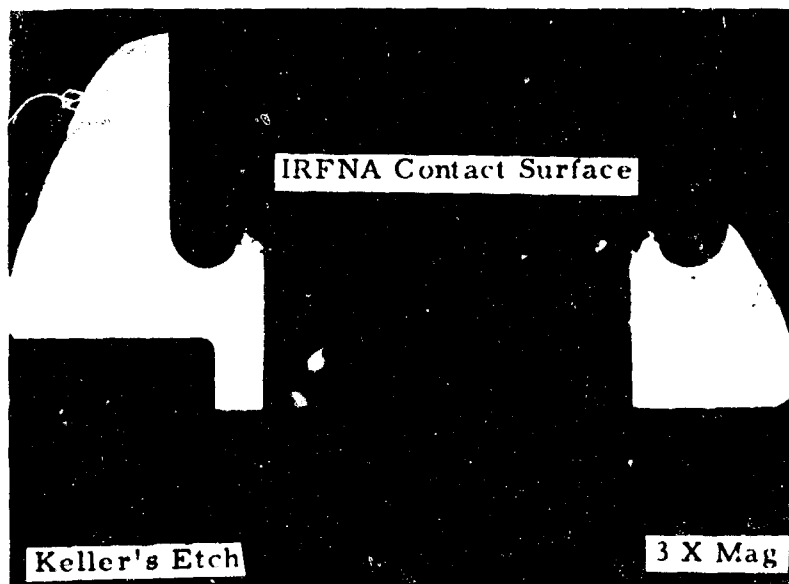
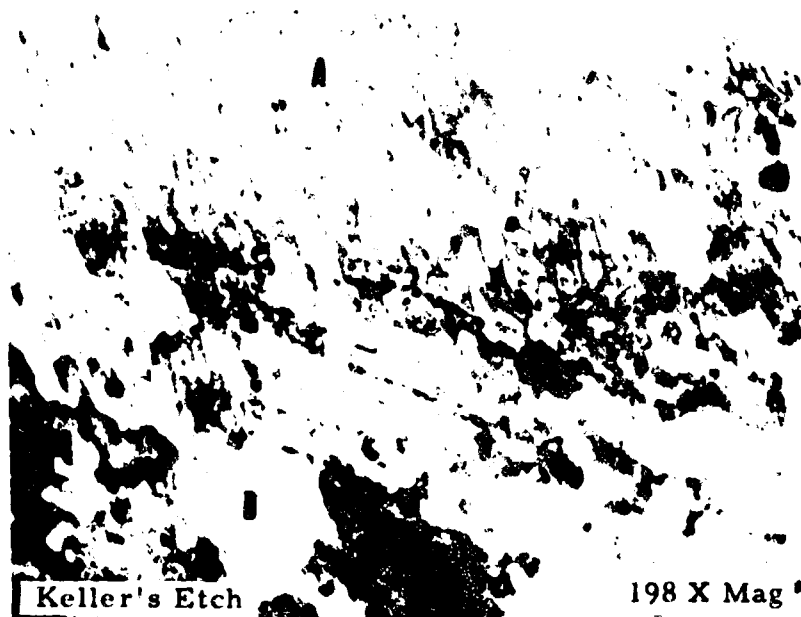


Figure 34. Oxidizer Orifice to Center Bulkhead
Shear Slide Housing Weld



Keller's Etch

198 X Mag

- A. Weld Metal
- B. Base Metal (Orifice)

Figure 35. Oxidizer Orifice to Center Bulkhead Weld Fusion Zone



Keller's Etch

198 X Mag

- A. Weld Metal
- B. Base Metal (Orifice)

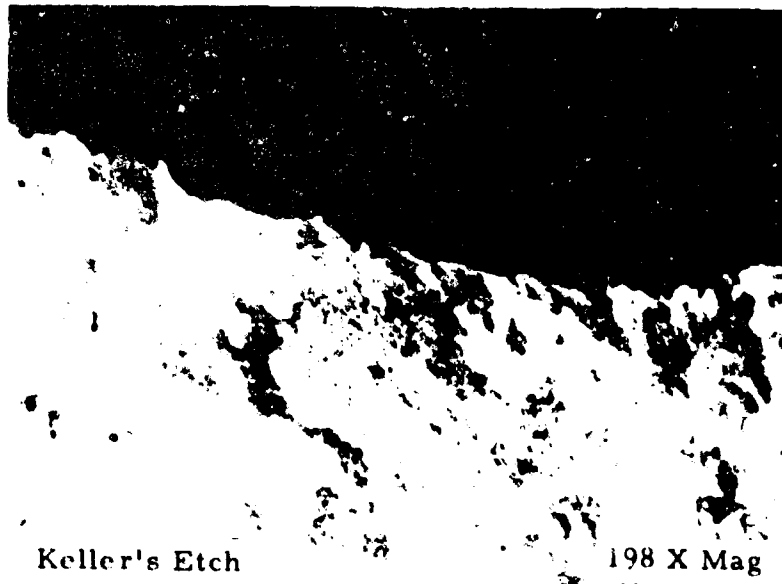
Figure 36. Oxidizer Orifice to Center Bulkhead Weld Fusion Zone



Keller's Etch

198 X Mag

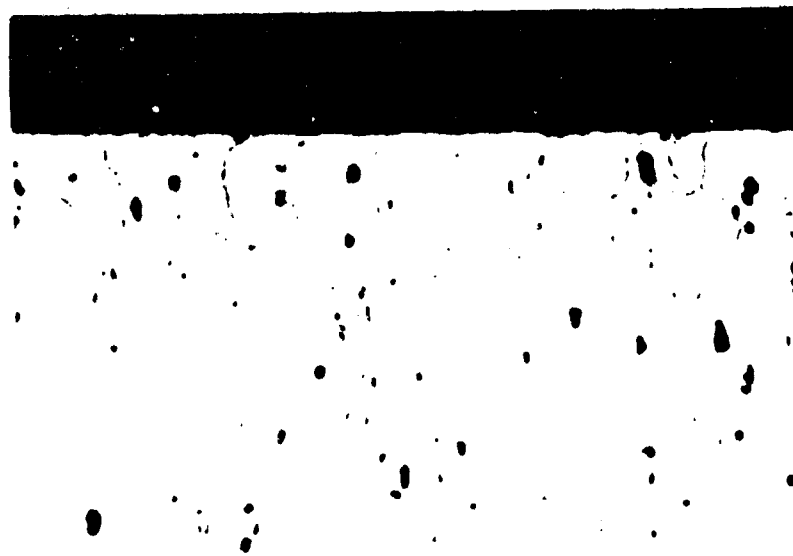
Figure 37. IRFNA Contact Surface, Center Bulkhead
HAZ at Orifice Weld



Keller's Etch

198 X Mag

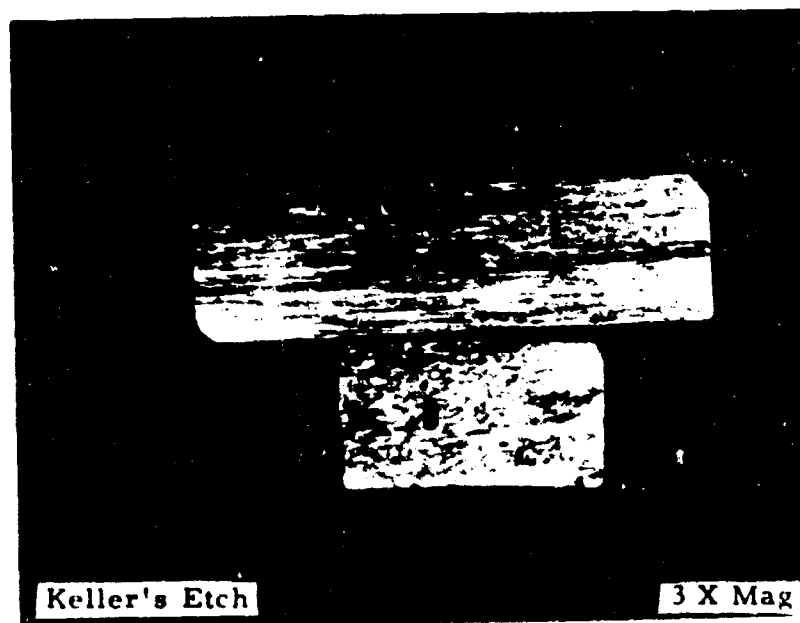
Figure 38. IRFNA Contact Surface, Shear Slide
Housing HAZ at Orifice Weld



Keller's Etch

198 X Mag

Figure 39. IRFNA Contact Surface, Oxidizer Orifice



Keller's Etch

3 X Mag

A. Cut Parallel to Centerline

B. Cut Perpendicular to Centerline

Figure 40. Aluminum Oxidizer Tank Wall



Keller's Etch

198 X Mag

Cut Parallel to Tank Centerline

Figure 41. IRFNA Contact Surface, Oxidizer Tank Wall

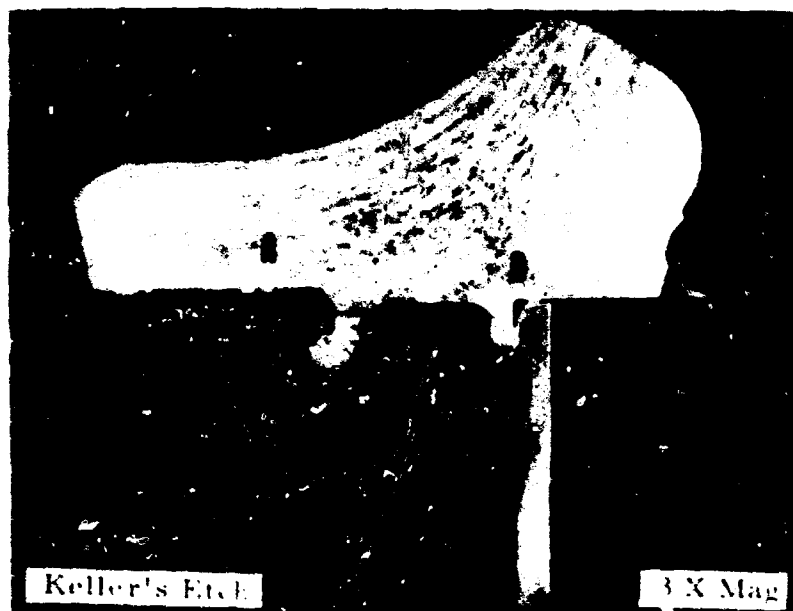


Keller's Etch

198 X Mag

Cut Perpendicular to Tank Centerline

Figure 42. IRFNA Contact Surface, Oxidizer Tank Wall



- A. Fuel Tank Burst Band
- B. Fuel Tank Baffle

Figure 43. Center Bulkhead Burst Band at Fuel Tank and Baffle Welds

Keller's Etch

3 X Mag

- A. Base Metal (Bulkhead)
- B. Weld Metal

Figure 44. Fuel Tank Deflector to Center Bulkhead Weld Fusion Zone

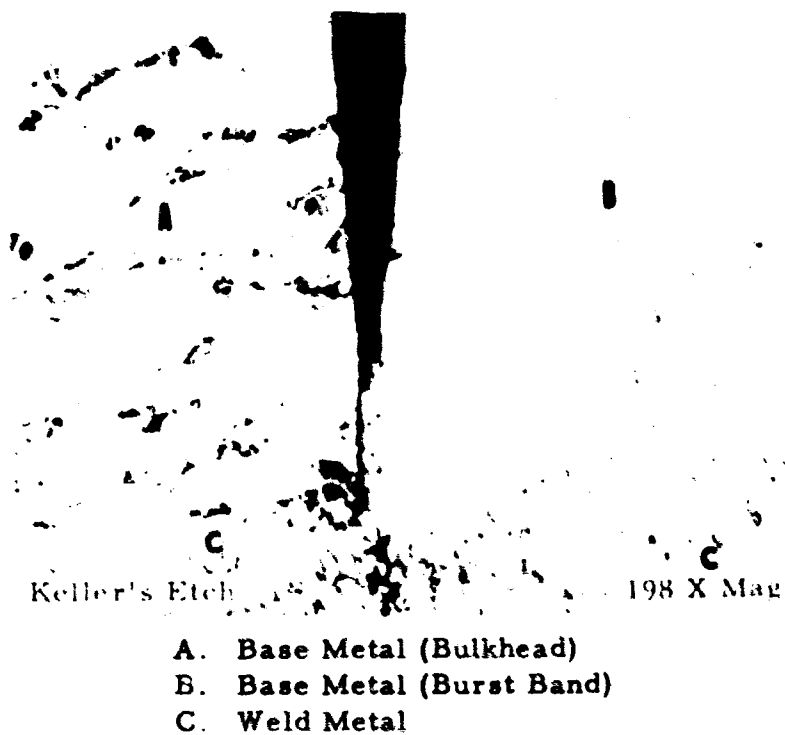


Figure 45. Fuel Tank Burst Band to Center Bulkhead Weld Fusion Zone



Corrosion is more advanced on this surface than on the IRFNA contact surfaces.

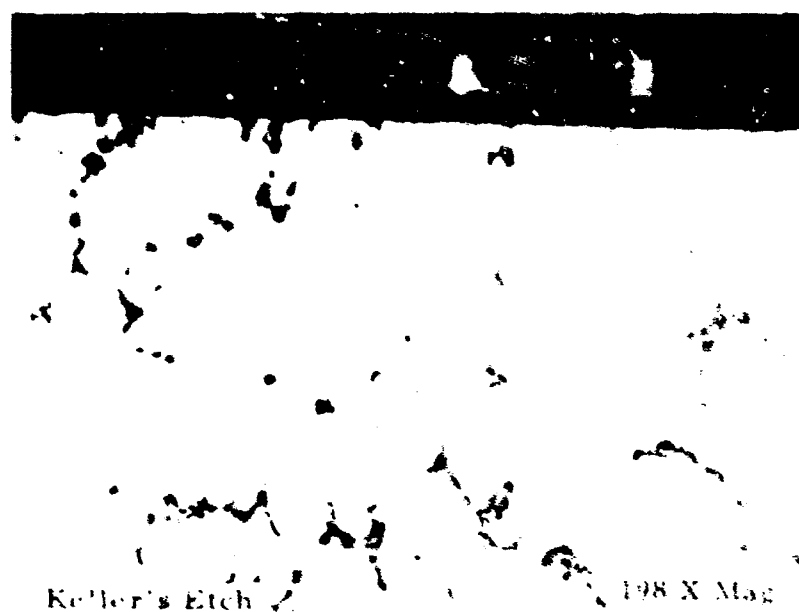
Figure 46. Atmosphere Surface, Center Bulkhead



Keller's Etch 198 X Mag

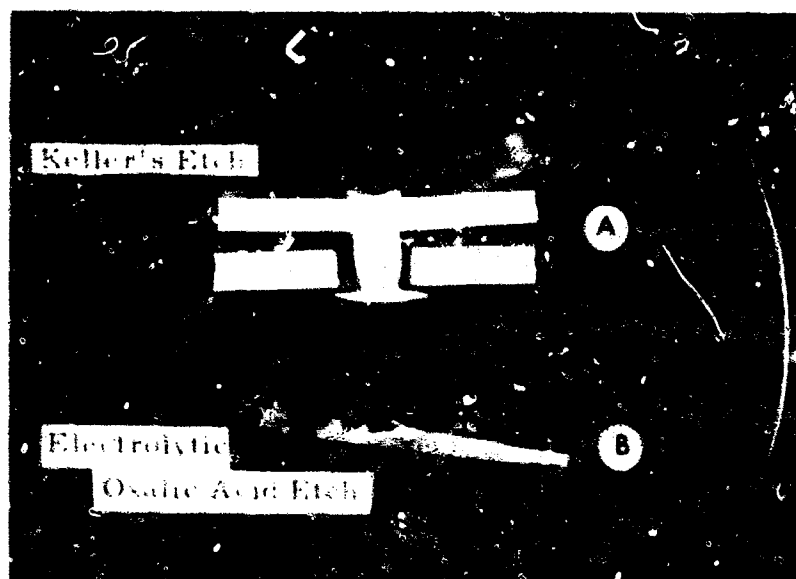
- A. Base Metal (Baffle)
- B. Weld Metal

Figure 47. Fuel Tank Baffle to Bulkhead Weld Fusion Zone



Keller's Etch 198 X Mag

Figure 48. Fuel Contact Surface, Center Bulkhead at Fuel Tank Baffle Weld HAZ



- A. Aluminum Retainer, Director, and Rivet;
Stainless Steel Screen
- B. Stainless Steel Retainer and Screen

Figure 49. Oxidizer Director and Screen Assembly

Keller's Etch

100 X Mag

Slight IRFNA attack apparent on heavily worked rivet [A].

Figure 50. Rivet Joint, Aluminum to Aluminum Contact, Oxidizer Director and Screen Assembly

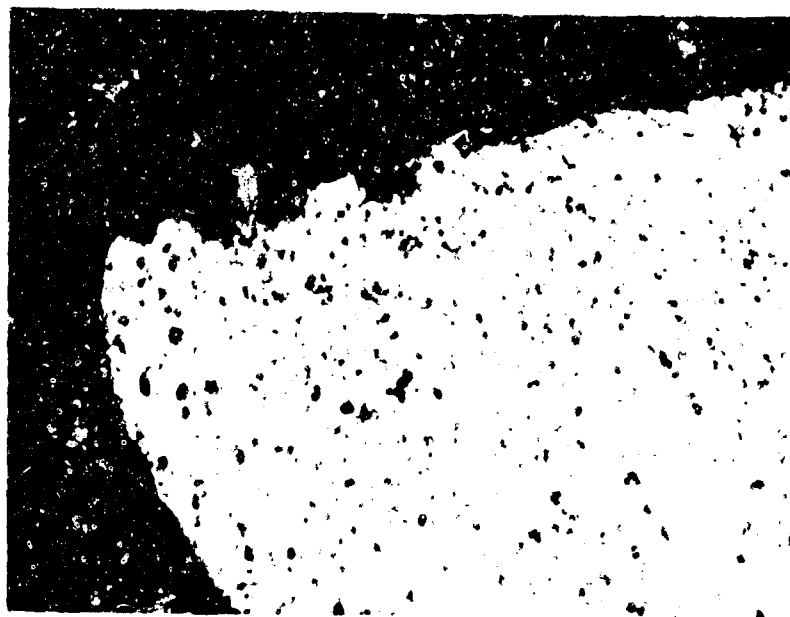
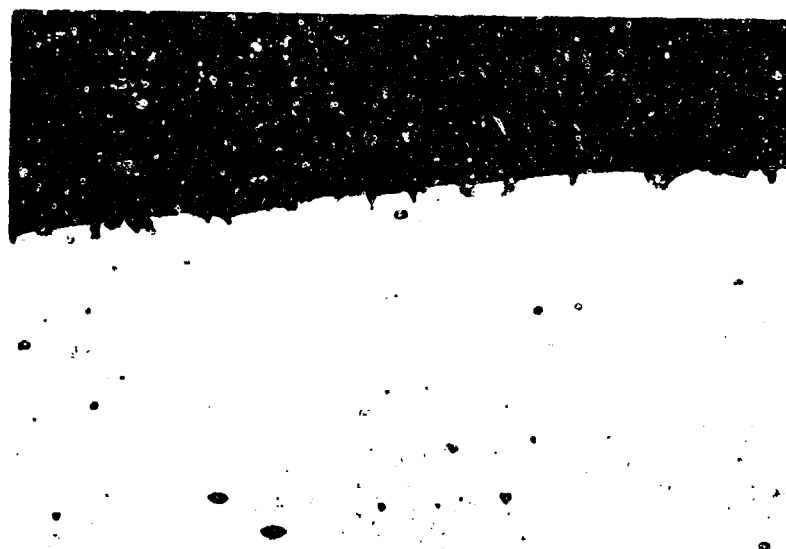


Figure 51. Corrosion on "Bucked" End of Rivet,
Oxidizer Director and Screen Assembly



Electrolytic Oxalic Acid Etch 198 X Mag
Slight IRFNA attack is evident.

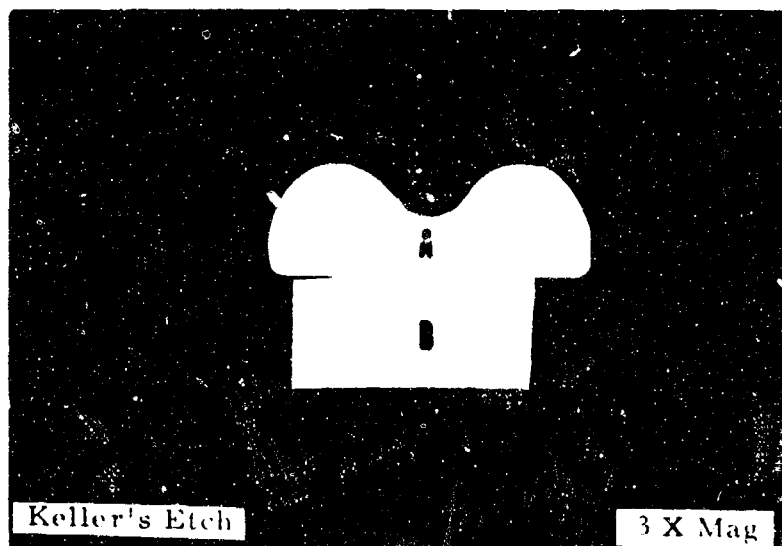
Figure 52. IRFNA Contact Surface, Stainless Steel
Screen



Electrolytic Oxalic Acid Etch 198 X Mag

No IRFNA surface attack is evident.

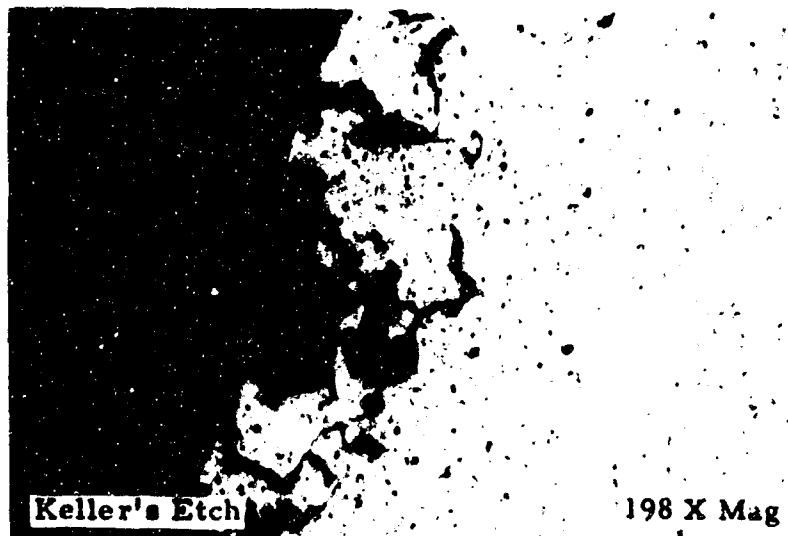
Figure 53. IRFNA Contact Surface, Stainless Steel
Outer Screen Retainer Ring



A. Transverse Section

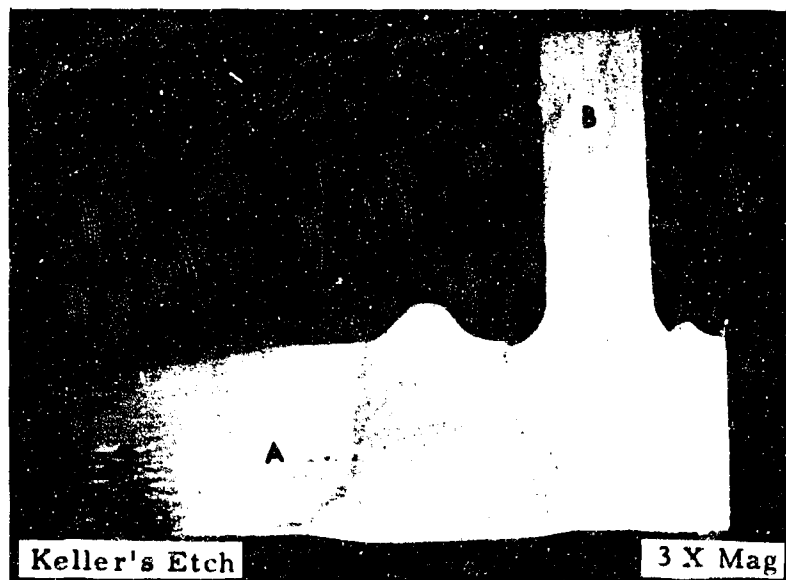
B. Longitudinal Section

Figure 54. Aluminum Retainer Ring, Oxidizer
Director and Screen Assembly



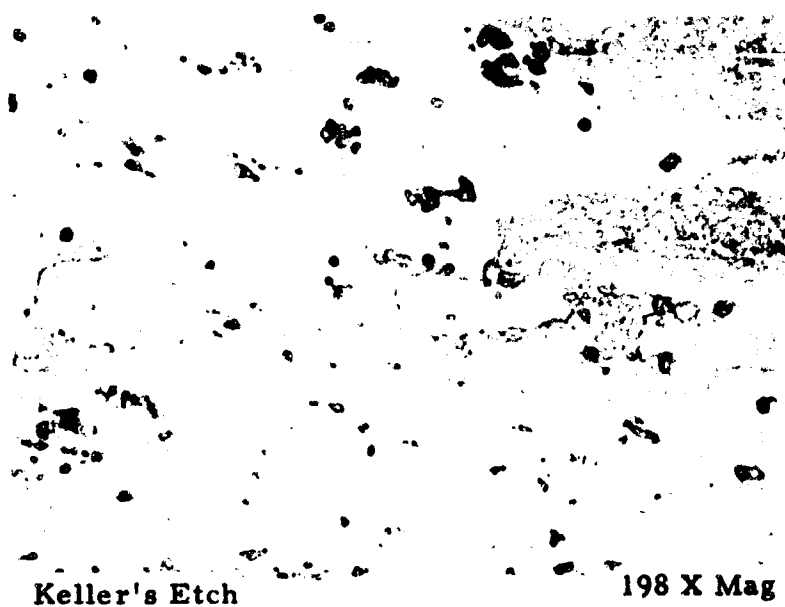
Corrosion has been advanced by exposure to laboratory environment.

**Figure 55. Corrosion Site on Retainer Ring
Where Aluminum Ring Contacted Stainless
Steel Screen**



A. Oxidizer Tank Wall
B. Bulkhead

Figure 56. Center Bulkhead to Tank Weld

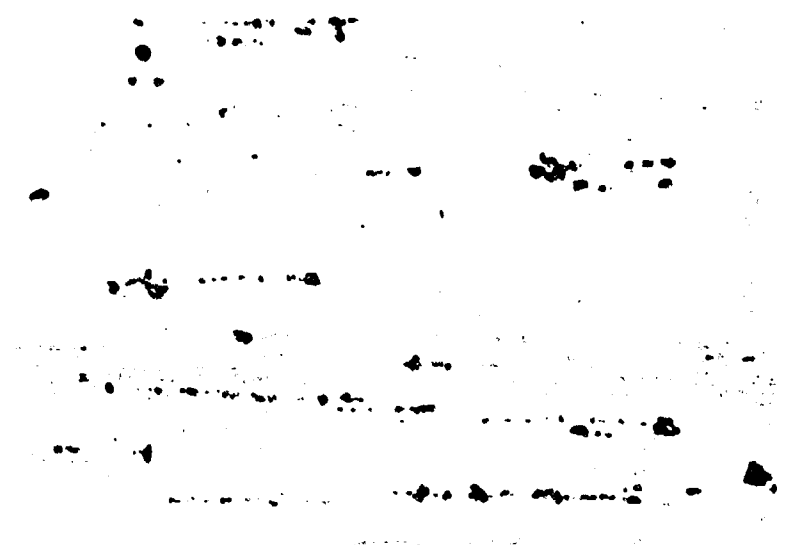


Keller's Etch

198 X Mag

Hardness, Rockwell "B" 80

Figure 57. Oxidizer Tank Wall Microstructure
12 Inches from Center Bulkhead Weld



Keller's Etch

198 X Mag

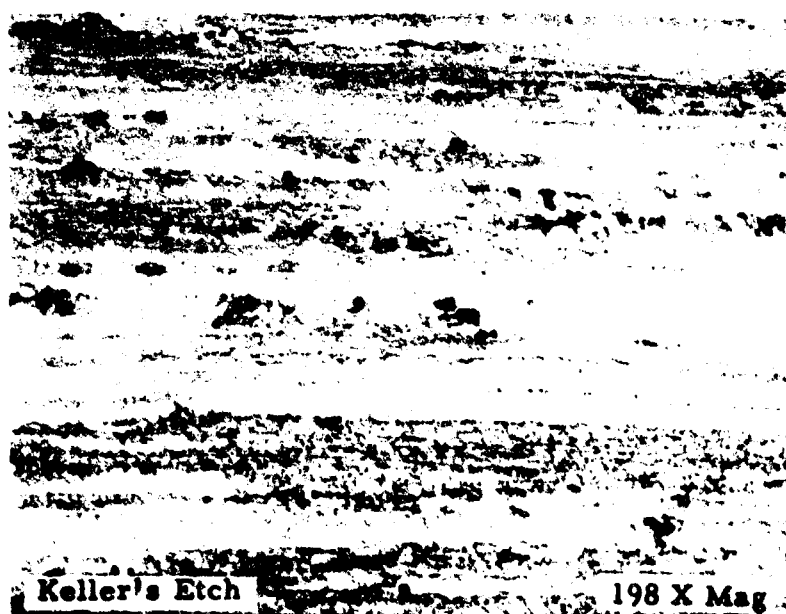
Hardness, Rockwell "B" 73.0

Figure 58. Oxidizer Tank Wall Microstructure
0.660 Inch from Center Bulkhead Weld



Hardness, Rockwell "B" 50.0

Figure 59. Oxidizer Tank Wall Microstructure
0.330 Inch from Center Bulkhead Weld



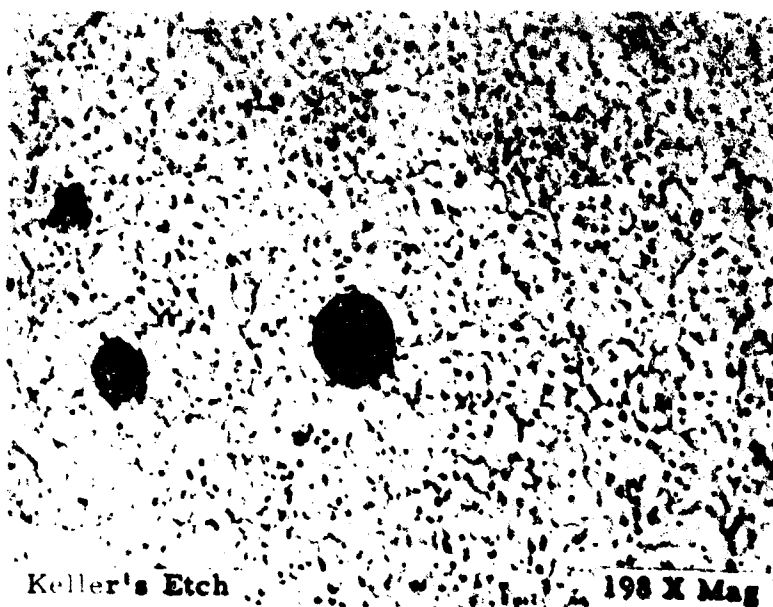
Hardness, Rockwell "B" 60

Figure 60. Oxidizer Tank Wall Microstructure
0.050 Inch from Center Bulkhead Weld



- A. Wall Material
- B. Weld Metal

Figure 61. Oxidizer Tank Wall to Center Bulkhead Weld



Hardness, Rockwell "B" 47.0

Figure 62. Weld Metal at Oxidizer Tank to Center Bulkhead Weld

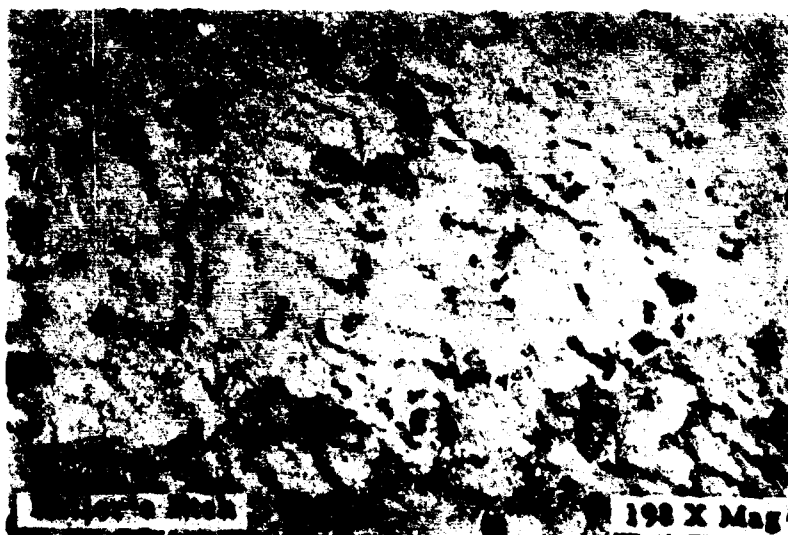


Hardness at bulkhead side of fusion line is approximately Rockwell "B" 60.

A. Bulkhead

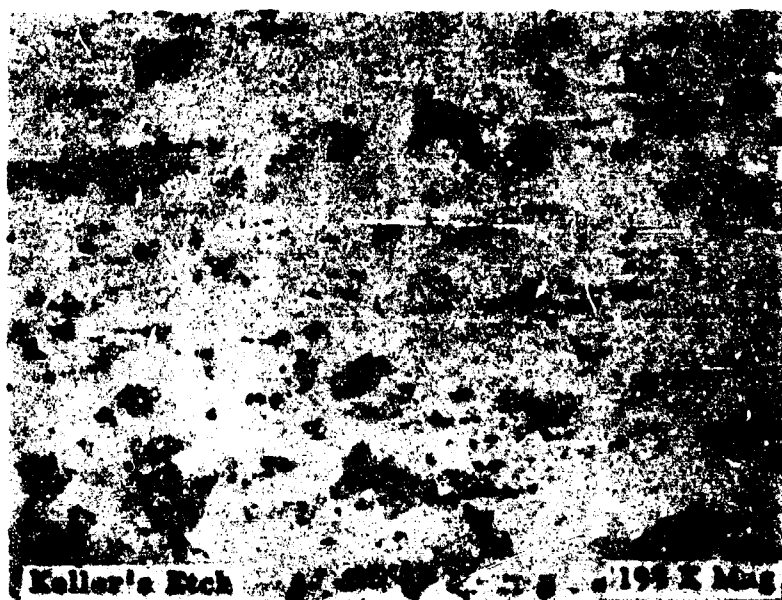
B. Weld Metal

Figure 63. Center Bulkhead Weld Fusion Zone.



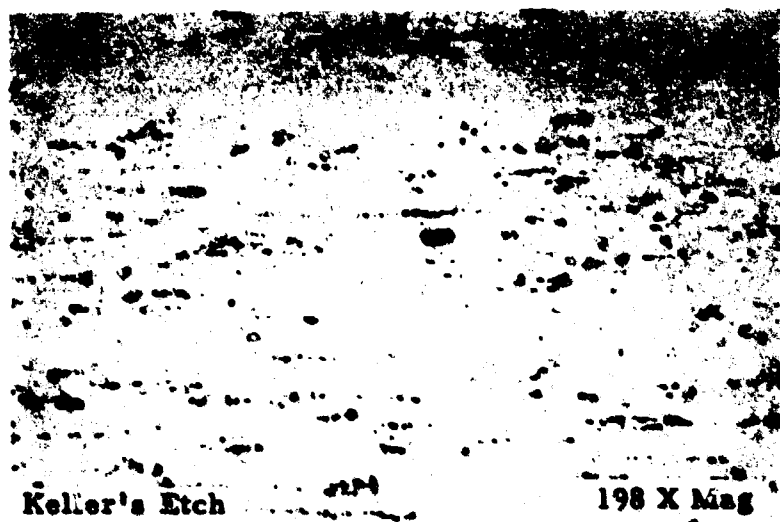
Hardness, Rockwell "B" 35.5

Figure 64. Center Bulkhead Microstructure 0.180 Inch from Both Oxidizer Tank Weld and Fuel Tank Weld



Hardness, Rockwell "B" 53.0

Figure 65. Center Bulkhead Microstructure 0.500
Inch from Weld



Hardness, Rockwell "B" 71.8

Figure 66. Center Bulkhead Microstructure 0.75
Inch from Weld

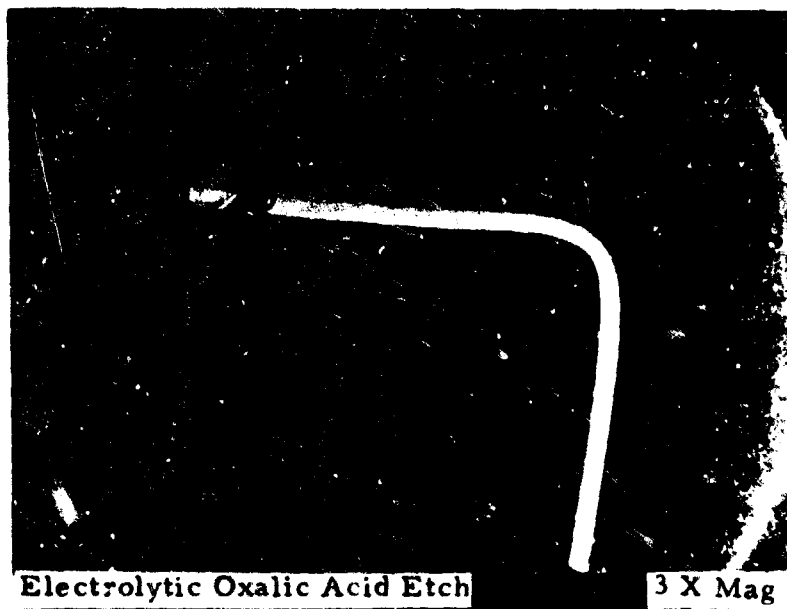
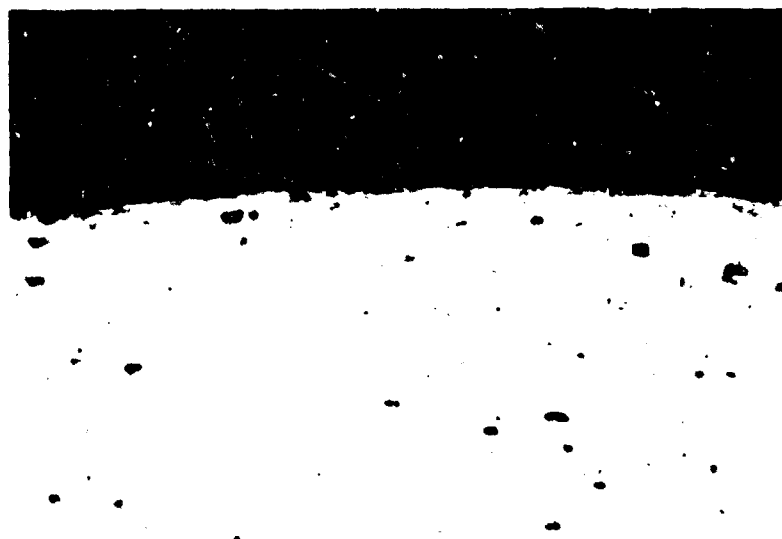


Figure 67. Stainless Steel Solid Propellant Gas Diffuser and Deflector, Photomicrograph



Electrolytic Oxalic Acid Etch 198 X Mag

No corrosive attack is evident.

Figure 68. Stainless Steel Solid Propellant Gas Diffuser and Deflector, Microstructure



Figure 69. Fuel Tank, Aft End, Sectioned View

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R&D		
<small>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</small>		
1. ORIGINATING ACTIVITY (Covering entire report) Structures and Mechanics Laboratory Research and Development Directorate U.S. Army Missile Command Redstone Arsenal, Alabama 35809		2a. REPORT SECURITY CLASSIFICATION Unclassified Report
		2b. GROUP N/A
3. REPORT TITLE METALLURGICAL EXAMINATION OF BULLPUP "A" PROPULSION UNIT S/N 4800 AFTER THREE YEARS OF STORAGE		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) None		
5. AUTHOR(S) (Last name, first name, initial) Davidson, James T.		
6. REPORT DATE 28 April, 1966	7a. TOTAL NO. OF PAGES 48	7b. NO. OF REFS None
8a. CONTRACT OR GRANT NO. A. PROJECT NO. (DA) 1X242403D231	9a. ORIGINATOR'S REPORT NUMBER(S) RS-TR-66-5	
	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) AD	
10. AVAILABILITY/LIMITATION NOTICES This document is subject to special export controls, and each transmittal to foreign governments or foreign nationals may be made only with prior approval of this command. Attn: AMSMI, RSM.		
11. SUPPLEMENTARY NOTES N/A	12. SPONSORING MILITARY ACTIVITY Same as No. 1	
13. ABSTRACT A tactical Bullpup "A" prepackaged, storable, liquid propulsion unit was metallurgically examined after three years of storage. The examination was conducted relative to the storage requirements of the LANCE missile system (currently being developed by the U.S. Army Missile Command, Huntsville, Alabama) which utilizes materials and propellants similar to those employed in the BULLPUP "A". The purpose of the evaluation was to acquire "real-time" storage data based on long periods of exposure. The examination revealed that containment of MAF-1 fuel and inhibited red fuming nitric acid oxidizer by 2014-T6 aluminum alloy tankage for more than three years had not affected the mechanical, physical, or chemical properties of the materials to a degree that would affect tactical performance.		

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